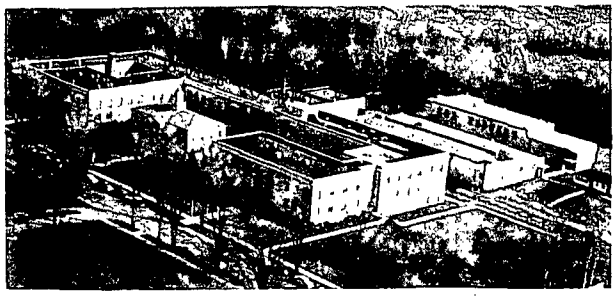

THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

# DEVELOPMENT OF AN IMPROVED DIFFUSION BOARD MATERIAL

Project 2256

Report Sixteen

An Annual Report

to

U. S. ARMY CHEMICAL CENTER PROCUREMENT AGENCY

Report Period: October 4, 1960 to October 4, 1961

April 1, 1962

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

# DEVELOPMENT OF AN IMPROVED DIFFUSION BOARD MATERIAL

Project 2256

Contract No. DA18-108-405-CML-941
DA18-108-CML-6561
Order No. CP 1-405-4519

Report Sixteen

Annual Report

to

U. S. ARMY CHEMICAL CENTER PROCUREMENT AGENCY

Report Period: October 4, 1960 to October 4, 1961

April 1, 1962

TABLE OF CONTENTS


| | Page |
|---|---|
| SUMMARY | 1 |
| PROCESS STUDY | 3 |
| Laboratory Forming and Testing Procedures | 3 |
| Forming of Boards | 3 |
| Caliper and Density | 4 |
| Carbon Dioxide Diffusivity | 4 |
| D.O.P. Smoke Penetration | 5 |
| Gas Life | 5 |
| Charcoal Loading | 6 |
| Mildew Resistance | 6 |
| Water Absorption | 7 |
| Strength Tests | 8 |
| Accelerated Aging | 9 |
| Laboratory Production of Existing Board | 9 |
| Pulps Used | 10 |
| Minnesota and Ontario Pulps | 11 |
| Repulped Newspaper | 14 |
| Mosinee Kraft | 14 |
| Wood Conversion Company Pulps | 15 |
| Armstrong Cork Company Pulps | 16 |
| General Comparison of Pulps | 16 |
| The Use of Sizing and Wet-Strength Agents | 17 |
| Aquapel | 17 |
| Kymene 557 | 22 |
| Cyron Size | 22 |
| Isothermal Moisture Absorption | 26 |
| The Use of Fungicides | 29 |

Page

Effects of Planing 30

The Use of Stabilizers 31

The Variables Affecting Gas Life 32

Effect of pH 32

The Critical Bed Concept 33

Aging 35

Mechanism of CK Protection 37

PILOT TRIALS 38

PRODUCTION PLANNING 41

Drying Studies 41

Flammability 41

Ignition Temperature 41

Flame Test 42

Negotiation for Production Run 43

GENERAL DISCUSSION AND PRESENT STATUS 44

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

# DEVELOPMENT OF AN IMPROVED DIFFUSION BOARD MATERIAL

## SUMMARY

This program has for its objective the development of an improved diffusion board material. In a previous program a diffusion board was developed which satisfactorily met the requirements of the Chemical Corps with respect to gas life, aerosol penetration, and diffusional characteristics but was limited to indoor use because it lacked weatherability and other physical properties necessary for field use. The present contract was directed toward the development of a board capable of both indoor and outdoor use by improving such characteristics as water resistance, scuff resistance, bonding, mildew resistance, tensile strength, and durability without major detriment to the protective characteristics of diffusion, resistance to aerosol penetration, and gas protection.

The contract included a process study on the diffusion board material as already developed and on methods for improving it and an agreement to subcontract with commercial organizations for one or two limited production runs on reduced scale equipment and one (two if needed) production run of 15,000 sq. ft. on full-scale equipment.

Following approval of the contract, reports of previous work were received and reviewed and the entire program discussed with Army Chemical Center personnel. Protective properties were defined as: 1. Adequate diffusion characteristics, measured as carbon dioxide diffusion and being as high as possible but not less than 0.02 sq. cm. per second; 2. Resistance to aerosol penetration, as measured by the DOP test with a specification of not over 0.01% penetration under the test conditions described; and 3. A minimum gas life of 20 minutes under the test conditions described in the contract. Previous work indicated that quarter-inch

board at a density of 21 lb. per cu. ft. and an ASC charcoal content of 5.0 grams/100 sq. cm. gave good results. Physical properties of interest included tensile strength, both dry and after water exposure, and water repellency. Test methods for these properties had not been chosen. Other factors of importance were resistance to mold growth and retention of gas life under various conditions of exposure, but no specific methods or specifications had been chosen for these aspects.

The process study was initiated and carried out as described in this report. A promising pilot-scale run was made and plans were initiated for the full-scale production runs. At this time the serious deterioration of CK life upon exposure of diffusion board to tropical aging conditions was noted and arrangements for the production runs were postponed. Alternate proposals were submitted with the choice based on the relative importance of CK life and tropical aging; one covered a production run of board on the basis of the performance obtained to date and the other proposed a more thorough study of the mechanism of CK protection and the factors governing it before proceeding with additional development work. The Institute of Paper Chemistry was then requested to continue the process study and to subcontract for two production runs, improving the general properties as much as possible without sacrificing any of the protective characteristics obtained to date. A contract for extension of the original program was approved on February 13, 1962 and work on this extension has been initiated. As part of that extension the preparation of a termination report was delayed until the completion of the extended contract, and an annual report was requested in its place. This report includes a summary of the work included in the first one-year program. Further details may be found in the monthly reports as indicated.

## PROCESS STUDY

### LABORATORY FORMING AND TESTING PROCEDURES

#### Forming of Boards

After preliminary trials, boards were formed in a 14-1/2 by 14-1/2-inch Valley sheet mold on a 16-mesh bronze wire screen. The standard procedure for draining the mold was to drain through a water-leg of approximately 3 feet until the forming water level was even with the upper surface of the formed board and then to apply a 20-25 inch mercury vacuum to complete the drainage. Late in the program a brass baffle was installed beneath the wire in order to reduce the turbulence encountered during draining and to improve the board formation.

The procedure for producing a board was to:

1. Slurry enough fiber for one board at a consistency of 4% with a Lightnin' mixer (exceptions being the boards formed from repulped newspaper and some of the first boards formed from Minnesota and Ontario wet lap which were slurried in a Somat Repulper).

2. Add charcoal and other additives (some sizing materials were added in the deckle box; however, these deviations from procedure were noted), note pH and adjust, if necessary.

3. Pour slurry into the deckle box, dilute to 1%, (this dilution was reduced to 0.7% late in the program), stir, note pH and adjust if necessary, and form.

4. Remove the pad and screen, and press for ten minutes in hydraulic press at pressure required to produce 20 to 21 lb./cu. ft. board, usually 150 p.s.i.

5. Place in circulating-air electric oven set at 105°C. for three hours. In the early phases of the program the boards were dried for two hours; however,

it was found that the board contained sufficient moisture at the end of two hours to be detrimental to gas life.

The charcoal used in the production of all laboratory boards was charcoal ASC Grade 1 through 140 mesh, manufactured according to SPEC MIL-C-13724, Lot NY 5181-5 Pkg. No. 248, packaged in August, 1955.

In making additions to the pulp slurries, a period of five minutes was allowed between additions in order to insure good mixing. Twenty-five ± 5 minutes was allowed between the addition of charcoal and pressing in order to mitigate any variant effects on the activity of the charcoal due to leaching. The standard addition of charcoal used was 25% based on ovendry fiber.

Caliper and Density

Caliper values were reported as the average of ten individual measurements taken with a Cady automatic micrometer, recorded to the nearest thousandth of an inch.

Densities were determined under ambient conditions, using the average caliper and the average of three length and three width determinations taken to the nearest 1/32 of an inch, and the weight of the specimen to the nearest 0.1 gram.

Carbon Dioxide Diffusivity

The diffusivity was measured as the change in concentration of carbon dioxide from an initial concentration of 2% to 50% over a period of 20 minutes resulting from diffusion of the carbon dioxide through a 12 by 12-inch section of a specimen. The apparatus was constructed of metal and designed according to information received from the Army Chemical Center. It consisted of a 12 by 12 by 12-in. box with an open [illegible] clamps for attaching a 14 by 14-in.

specimen. Carbon dioxide concentrations were measured with an Orsat gas analyzing apparatus. Results were reported as sq. cm./sec. (actual units: cc./cm./sec.) calculated from the following derived relation:

$$\underline{D} = \frac{(70.1)\ (\underline{l})\ (\log \underline{C_1}/\underline{C_2}}{\underline{t}}$$

where:

$\underline{D}$ = Diffusivity in sq. cm./sec.

$\underline{l}$ = Thickness of the specimen in cm.

$\underline{C_1}$ = Initial $CO_2$ concentration, % by volume.

$\underline{C_2}$ = Final $CO_2$ concentration, % by volume.

$\underline{t}$ = Time, sec.

D.O.P. Smoke Penetration

Smoke penetration was determined with the Naval Research Laboratory's Dioctyl Phthalate smoke penetration apparatus in accordance with the procedure outlined by the Army Chemical Center as the per cent of smoke passing through a specimen at a flow rate of one liter per minute.

Gas Life

Gas life determinations were run by the Army Chemical Center with two gases: cyanogen chloride and chloropicrin. The cyanogen chloride test results were reported as the number of minutes of protection given by 100 sq. cm. of sample (conditioned to 80% R.H., 80°F.) against a gas concentration of 4 mg. per liter at 80% R.H., 80°F. at a test flow of one liter per minute. At first, the gas lives were corrected to a standard 5 g./100 sq. cm. charcoal loading (based on ash tests and assuming a straight-line correction); later the CK gas life data were converted to a critical bed depth value derived in a plot of CK gas life

versus charcoal loading as the zero gas life intercept of a line having a slope of 26 [illegible] min./g./100 sq. cm. The chloropicrin test results were reported as the number of minutes of protection provided by a 100-sq. cm. specimen tested as received, against a gas concentration of 50 mg. per liter at 50% R.H. at a flow rate of one liter per minute.

Charcoal Loading

Charcoal retentions were determined by the difference in the ash content of the charcoal-loaded specimen and a blank specimen on the basis of the ash content of the charcoal. In order to correct for loss of material through leaching, ash [illegible] of charcoal samples dispersed in water for varying periods of time were determined and the comparable ash content used according to the charcoal-water contact time involved in producing the board tested. Ashing was carried out at 900°C. according to Institute Method 422. Calculations were on the basis of oven-dry [illegible] weights.

The total charcoal-liquid water contact time involved in the production [illegible] board sample was approximately one-half hour. Consequently, the [illegible] dispersed in water for one-half hour, 29.60%, was used in [illegible] calculations. The following equation was used in the calculations:

$$\% \text{ charcoal} = \frac{b - a}{c - a}$$

where: $a$ = % ash in a blank (board containing no charcoal)

$b$ = % ash in the sample

$c$ = % ash in charcoal corrected for contact time with water.

Preservatives

Preservatives were evaluated by determining the [illegible] of [illegible] and [illegible] Aspergillus globosum over a period of five [illegible]

specimens were soaked in a mineral salts solution, placed in contact with an agar medium and inoculated with a spore suspension. This test was a more drastic modification of the procedures outlined in the Federal Specification Number CCC-T-191b, Method 5751 "Mildew Resistance of Cloth; Direct Innoculation, Pure Culture, Non-Sterile Specimen Method." (By the original method, no growth was observed on board samples without fungicides added.)

Water Absorption

Water absorption characteristics of board samples were evaluated by determining the water absorbed by samples submerged under a one-inch head of water for 2 hours and 24 hours. Specimens, 6 by 6 inches, were used for the tests and were tested by the following procedure:

The specimens were dried for 24 hours at 160°F. and cooled to room temperature in a dry atmosphere (desiccator jar). The specimens were weighed, calipered, and submerged under 1 inch of distilled water maintained at 70 $\pm$ 5°F. After two hours' submersion, each specimen was removed from the water, placed on edge to drain for 10 minutes, carefully blotted with blotting paper and weighed. After weighing, each specimen was resubmerged for a period of 22 hours and reweighed in the same manner. The volumes of water absorbed were calculated and the water absorption was reported as per cent by volume of water absorbed based on the dry volume.

References for these tests were the Federal Specification LLL-I-535 "Federal Specification for Insulating Fiberboard" and ASTM D 1037-60T, "Tentative Methods of Test for Evaluating the Properties of Wood-Base Fiber and Particle Panel Materials."

Strength Tests

Tensile and transverse strength tests were run according to ASTM C-209-60 "Testing Structural Insulating Board Made From Vegetable Fibers." Three specimens cut in the machine direction and three specimens cut in the cross-machine direction of each sample were used for each test. All of the specimens were conditioned to 50 ± 5% relative humidity, 72 ± 2°F. with the exception of the wet tensile specimens. Wet tensile tests were run on specimens after 2 hours soaking under 1 inch of distilled water at 72 ± 2°F. and after 24 hours of soaking. Dry tensile tests were run on conditioned samples and samples reconditioned after 24 hours of soaking.

Tensile tests were run on dumbbell-shaped specimens 10 inches in overall length, 2 inches maximum width, necked down to 1-1/2 inch minimum width along a 2 inch flat in the center. Rate of jaw separation was set at 0.5 in./min. (ASTM specifies 2 in./min. but this was found to be too rapid for the response of the machine.) Specimens breaking within 1/2 inch of the jaws were discarded. Cross-sectional area at the point of break was determined and maximum tensile strength was reported in p.s.i.

Transverse strength tests were run on 3 by 12-in. specimens loaded at mid-span on bearings 12 inches apart. All bearing edges were 3/8-inch radius. Loading was applied at a rate of 5 in./min. until failure. Results were reported as maximum load, deflection at breaking point, and modulus of rupture. The modulus of rupture was computed by the formula given in the TAPPI Standard T 1003 sm-55 "Flexural Resistance and Deflection of Insulating Fiberboard":

$$R = \frac{1.5\ P\ L}{W\ (\ )^2}$$

where:

R = Modulus of rupture, p.s.i.

P = Loading, lb.

L = Span length, inches

W = Width, inches

t = Thickness, inches

Accelerated Aging

Accelerated aging was carried out at the Army Chemical Center. Two conditions were used; desert and tropical. Boards aged under tropical conditions were equilibrated at 80°F., 80% R.H. and stored in sealed containers for one week at 113°F. Desert aging consisted of two weeks storage at 160°F., specimens being conditioned to less than 1% moisture, by weight, at the start of the test.

LABORATORY PRODUCTION OF EXISTING BOARD

The diffusion board developed and used prior to this program was reported to possess the following technical characteristics:

Caliper -- 1.23 in. minimum
1.27 in. maximum
1.25 in. desired

Density -- 19 lb. per cu. ft. minimum
23 lb. per cu. ft. maximum
21 lb. per cu. ft. desired

ASC charcoal content -- 4.5 grams per 100 sq. cm. minimum
5.5 grams per 100 sq. cm. maximum
5.0 grams per 100 sq. cm. desired

Tensile strength -- approximately 100 lb. per in. of width (equivalent to 400 lb. per sq. in. for 1/4-in. board)

Board conforming to these characteristics had been produced commercially using 90% "Insulite" pulp (Minnesota and Ontario Paper Company) and 10% paper mill groundwood with an addition of 25% charcoal and fresh water in a pH range of 6.0 to 8.0. The first step in the present program was to determine the conditions required in the laboratory to produce board with physical characteristics conforming to the limits set in the contract. A quantity of Insulite pulp was obtained from the Minnesota and Ontario Paper Company in wet lap form and used to make boards with a range of pressing and drying conditions as described in Report 5. These boards were tested for density, carbon dioxide diffusivity, and smoke penetration, both at the Institute and at the Army Chemical Center. From these tests it appeared that a pressing condition of 10 minutes at 100 p.s.i. would yield a board with approximately the correct density and with a diffusion of approximately the right level or slightly below it (without carbon). Since it was known that the addition of carbon tended to improve the diffusion it appeared that there would be no difficulty in meeting the requirements of density, diffusivity, and smoke penetration.

For [illegible] with the addition of 25% ASC Grade I charcoal. The addition of [illegible] resulted in an increase in diffusivity but resistance to smoke penetration appeared to be satisfactory. First tests of gas life indicated results in the [illegible] minutes and up, suggesting that there would be no difficulty in producing a laboratory board equivalent in properties to the board which had been developed previously. Accordingly, the investigation of variables necessary for the [illegible] additional properties was initiated.

PULP USED

The various pulps that have been used and evaluated are described in [illegible]. The [illegible] suggest considerations in choosing a pulp for laboratory

and pilot use in evaluating various additive materials for the improvement of diffusion board has been the availability of the pulp from a commercial operation in an unsized form. The use of commercially produced pulps was considered desirable in order to maintain a closer relation between material produced in the laboratory and the product of a commercial operation. Comparisons are shown in Tables I and II.

Minnesota and Ontario Pulps

Two types of pulp were received from this company; a sample of white board wet lap taken from their commercial board operation (Report 2) and a sample of paper mill groundwood (Report 9). The white board wet lap contained a small amount of wax-rosin size; we also received a small amount of unsized white board wet lap which had been dewatered by hand for comparison with the sized lap.

The white board stock is similar to that used (with 10% of groundwood) in the production runs of diffusion board prior to the present program. Early use of this pulp in the laboratory was to set up conditions for forming, pressing, and drying that could be used throughout the project (Report 3). This pulp was used to determine the effects of forming pH on gas life (Report 5); the effects were particularly serious at pH levels below 7, consequently ruling out the use of rosin-alum sizing or any sizing under acid conditions. The pulp was washed with hot water in an effort to remove most of the wax-rosin sizing; the pulp, which had a pH of 5, was washed until the pH of the squeezings from a sampling was 8 (Report 4). Evidently, washing removed the unreacted alum but very little of the rosin as the water absorbed by board made from this pulp was less than that by board made from the unsized specimen. After some trials in which other sizing agents were incorporated, it was decided that further investigations on sizing using this pulp would be of limited value because of the presence of the

TABLE I

COMPARISON OF PULPS (UNSIZED AND UNAGED)

| Sample 2256- | Pulp | pH[a] | Canadian Freeness, cc. | Density, lb./cu.ft. | $CO_2$ Diffusivity, sq.cm./sec. x $10^{-2}$ | Smoke Penetration, 1 l./min. flow, $\Delta P_1$ mm. $H_2O$ | % | Gas Life: Chloropicrin, min. | Gas Life: Cyanogen Chloride, min. | Charcoal Loading, g./100 sq.cm. | Critical Bed Loading (CK), g./100 sq.cm. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 17-3 | Minnesota & Ontario white board wet lap (wax-rosin sized at mill) | 5.2 | 645 | 21.8 | 2.33 | | <0.001 | 36.0 | 33.1 | 5.2 | 4.8 |
| 22-1 | Minnesota & Ontario white board wet lap, unsized | 7.2 | 575 | 21.95 | 2.44 | ~20 | <0.001 | 33.3 | 41.4 | 4.1 | 2.5 |
| 30-1 | Minnesota & Ontario white board wet lap, hot water washed (size removal) | 8.3 | 640 | 21.25 | 2.56 | 18.5 | 0.0015 | 43.2 | 58.6 | 6.1 | 3.8 |
| 37-2 | Minnesota & Ontario spruce (groundwood) | 6.3 | 68 | 27.3 | 1.13 | >320 | -- | | 56.2 | 5.9 | 3.8 |
| 33-3 | Repulped newspaper | 5.6 | 345 | 22.9 | 1.86 | ~200 | <0.001 | 43.1 | 77.6 | 6.4 | 3.5 |
| 47-1[b] | Mosinee special kraft | 6.3 | 810 | 20.2 | 2.33 | | | 54.5 | 83.9 | 5.9[c] | 2.7 |
| 69-6 | Wood Conversion Co. 10-853 | 6.4 | 752 | 19.6 | 2.92 | 17.5 | <0.001 | 37.6 | 63.2 | 5.5 | 3.1 |
| 69-4 | Wood Conversion Co. 10-854 | 5.4 | 797 | 13.5 | | 5.5 | 0.014 | 24.6 | 5.0 | 4.8 | 4.6 |
| 69-2 | Wood Conversion Co. 10-855 | 6.1 | 731 | 22.3 | 2.47 | 29.0 | <0.001 | 29.3 | 20.3 | 5.6 | 4.8 |
| 72-1 | Wood Conversion Co. 10-890 | 5.9 | 755 | 20.7 | 3.13 | 16.0 | <0.002 | | 32.2 | 5.3 | 4.1 |
| 72-2 | Wood Conversion Co. 52.2% 10-855, 47.3% 10-853 | 6.5 | 740 | 21.4 | | 25.0 | <0.001 | 42.6 | 39.2 | 5.4 | 3.9 |
| P1-3-127 | Wood Conversion Co. Pilot run stock | | | | | | | | 62.0 | 5.7 | 3.4 |
| 17-4 | Armstrong pine (groundwood) | 6.3 | 743 | 18.4 | 3.86 | 12.5 | <0.001 | | 2.3 | 4.7 | 4.6 |
| 17-6 | Armstrong willow (groundwood) | 6.5 | 648 | 20.2 | 2.95 | 24.0 | <0.001 | | 16.6 | 5.6 | 4.9 |

[a] pH of the pulp as a 4% slurry in city water (pH 9).

[b] Wet-pressed 5 min. at 25 p.s.i.

[c] Based on assumed 20% charcoal content.

TABLE II

COMPARISON OF PULPS, SIZED AND UNSIZED--EFFECT OF AGING ON GAS LIFE (CK)

| Sample 2256- | Pulp | Sizing Added[a] | Cyanogen Chloride Gas Life, min. Unaged Specimen[b] | Ambient Storage, CK Life | Ambient Storage, days stored | Desert Storage, 2 weeks | Tropical Storage, 1 week | Charcoal Loading, g./100 sq.cm. Unaged Specimen[b] | Ambient Storage | Desert Storage | Tropical Storage | Critical Bed Loading, g./100 sq.cm. Unaged Specimen[b] | Ambient Storage | Desert Storage | Tropical Storage | Increase in Critical Bed as a Result of Aging, % Ambient Storage | Desert Storage | Tropical Storage |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| [illegible]-6 | Minnesota & Ontario white board wet lap, (wax-resin sized at mill) | No | 33.1 A | [illegible] | 136 | -- | -- | 5.2 A | 5.2 | -- | -- | 4.6 A | 5.0 | -- | -- | 4.2 | -- | -- |
| [illegible]-[illegible] | Minnesota & Ontario white board wet lap, not water washed | [illegible] | ~ 30 A | [illegible] | [illegible] | -- | -- | [illegible] A | [illegible] | -- | -- | ~ 4.7 A | ~ 5.2 | -- | -- | [illegible] | -- | -- |
| 22[illegible] | Repulped newspaper | No | 62.[illegible] A | [illegible] | [illegible] | -- | -- | 6.6 A | 6.6 | -- | -- | 4.0 A | [illegible] | -- | -- | 20.[illegible] | -- | -- |
| NP-3-122 | Repulped newspaper | No | 77.0 | -- | -- | 72.0 | 57.0 | 6.1 | -- | 5.2 | 5.5 | 3.2 | -- | 2.5 | 3.4 | -- | -21.8 | 6.2 |
| NP-[illegible]-122 | Repulped newspaper | No | 64.0 | -- | -- | 54.0 | 37.0 | 5.2 | -- | 5.0 | 5.8 | 2.9 | -- | 3.0 | 3.7 | -- | 3.5 | 27.6 |
| NP-2-122 | Repulped newspaper | Yes | 58.0 | -- | -- | 45.0 | 10.0 | 5.2 | -- | 5.3 | 5.4 | 3.0 | -- | 3.7 | 5.0 | -- | 23.4 | 66.7 |
| T8-3 | Wood Conversion Co. 52.2% 10-855; 47.8% 10-853 | No | 39.2 A | 18.0 | 116 | -- | -- | ~ 5.4 A | ~ 5.4 | -- | -- | 3.9 A | 4.7 | -- | -- | 20.5 | -- | -- |
| T8-21 | Wood Conversion Co. 52.2% 10-855; 47.8% 10-853 | Yes | 24.6 A | 3.0 | 110 | -- | -- | 5.7 A | 5.7 | -- | -- | 4.8 A | 5.6 | -- | -- | 16.7 | -- | -- |
| PR-3-127 | Wood Conversion Co. pilot run stock | No | 62.0 | -- | -- | 63.0 | 29.0 | 5.7 | -- | 5.6 | 6.1 | 3.4 | -- | 3.2 | 5.0 | -- | -5.9 | 47.0 |
| PR-4-127 | Wood Conversion Co. pilot run stock | No | 58.0 | -- | -- | 61.0 | 18.0 | 6.3 | -- | 5.4 | 5.9 | 4.1 | -- | 3.1 | 5.2 | -- | -24.4 | 26.8 |
| PR-1-127 | Wood Conversion Co. pilot run stock | Yes | 50.0 | -- | -- | 65.0 | 11.0 | 5.7 | -- | 6.2 | 5.8 | 3.3 | -- | 3.7 | 5.4 | -- | -2.6 | 42.1 |
| PR-2-127 | Wood Conversion Co. pilot run stock | Yes | 52.0 | -- | -- | 59.0 | 9.0 | 5.8 | -- | 5.9 | 5.7 | 3.8 | -- | 3.7 | 5.4 | -- | -2.6 | 42.1 |
| PRW-3-127 | Wood Conversion Co. pilot run stock, hot water washed | No | 76.0 | -- | -- | 64.0 | 37.0 | 5.3 | -- | 5.0 | 5.7 | 2.4 | -- | 2.6 | 4.3 | -- | 8.3 | 79.2 |
| PRW-4-127 | Wood Conversion Co. pilot run stock, hot water washed | No | 66.0 | -- | -- | 68.0 | 28.0 | 5.1 | -- | 5.0 | 5.2 | 2.6 | -- | 2.4 | 4.1 | -- | -7.7 | 57.7 |
| PRW-1-127 | Wood Conversion Co. pilot run stock, hot water washed | Yes | 60.0 | -- | -- | 64.0 | 7.0 | 5.2 | -- | 5.4 | 5.5 | 2.9 | -- | 3.0 | 5.2 | -- | 3.4 | 44.9 |
| PRW-2-127 | Wood Conversion Co. pilot run stock, hot water washed | Yes | 73.0 | -- | -- | 65.0 | 7.0 | 5.7 | -- | 5.1 | 5.3 | 2.9 | -- | 3.0 | 5.0 | -- | 3.4 | 37.9 |

[a]Sizing additives: 0.5% Aquapel 360; 0.2% Kymene 557 (active material based on ovendry fiber).

[b]Suffix A indicates less than 30 days' storage in ambient conditions.

rosin (Report 7). Since it was available, this pulp was used in the evaluation of fungicides, covered elsewhere in this report.

Repulped Newspaper

A few manufacturers use reprocessed paper for production of wallboard and this possibility seemed worthy of investigation. Newspaper was repulped in a Somat repulper and formed into boards according to the standardized laboratory method (Report 4). Some of this pulp was used to form board under acid conditions in order to confirm the work done with the Minnesota & Ontario white board to determine the effects of forming pH on gas life (Report 5).

Board formed under alkaline conditions from this pulp had good gas life characteristics (50 min. CK life); however, the density of the board formed was high and the carbon dioxide diffusivity was low. It was necessary to reduce the wet pressing conditions to 5 minutes at 25 p.s.i. in order to produce acceptable board. The high density and low diffusivity of the board produced from this pulp precluded any further use of it for experimental additive systems. However, it has been considered as a filler for the control of the density of board formed from other pulps.

Mosinee Kraft

A small sample of a special electrical-grade kraft pulp, produced by Mosinee Paper Mills Company was received. This pulp is produced commercially to have low ash and low content of soluble materials. Although this pulp might not be suitable for the production of low density board, it was a somewhat purer pulp than most and was considered for the evaluation of gas life effects.

As was the case with the repulped newspaper, it was necessary to reduce the wet pressing conditions to 5 minutes at 25 p.s.i. in order to produce acceptable

board samples. Board produced from this pulp had a very high gas life; chloropicrin life was 54 minutes and the cyanogen chloride life was 84 minutes in the sample tested. No further work was done with this pulp because of the small quantity available and its specialized nature.

Wood Conversion Company Pulps

Four samples of aspen pulp stocks used by the Wood Conversion Company were received (Report 7). These pulps were described as follows:

10-853 is No. 3 machine stock with a mill freeness of 700 cc. containing broke but no size. It is composed of 16% broke, 55% 700-cc. mill freeness cooked aspen, 19% 1300-cc. mill freeness raw aspen "D" fiber, and 10% 465-cc. mill freeness cooked aspen.

10-854 is aspen "D" fiber with a mill freeness of 1350 cc.

10-855 is cooked aspen slow stock which has been refined in a 416 Bauer refiner to a mill freeness of 500 cc.

10-890 is similar to the 10-855 stock except that it has not been refined in the Bauer refiner.

We also obtained from Wood Conversion Company pulp for the pilot run as described in that section.

Use of the individual pulps resulted in the formation of boards ranging in density from 13.5 lb./cu. ft. to 22.3 lb./cu. ft. The low density board was produced from the pulp sample (10-854) which was the freest of the samples; it was not considered of any value for use in diffusion board. From the densities of the boards produced from the individual samples, it was determined that a 21 lb./cu. ft. board could be produced from a mixture consisting of 52.0% 10-855 and

67.5% 10-853 or a mixture of 17% 10-855 and 83% 10-890. These two pulp mixtures were used in the evaluation of alkaline size and wet-strength agents as described in those sections.

Armstrong Cork Company Pulps

Samples of pine groundwood and willow groundwood pulps received from the Armstrong Cork Company were formed into boards to be evaluated in terms of gas life (Reports 7 and 9). Each of these pulps contained a small amount of Dowicide G preservative.

Board formed from the pine pulp was less dense (18.4 lb./cu. ft.) than the density desired in diffusion board. The willow pulp produced slightly denser board (20.2 lb./cu. ft.). Formation was poor in both cases; chips and large fibers were imbedded in the top surfaces of the boards, particularly in the boards from the pine groundwood.

General Comparison of Pulps

A comparison of the various pulps used in laboratory work is given in [illegible] and 2. There are palpable differences in pulps in terms of density, gas [illegible] penetration and carbon dioxide diffusivity. The gas life seems to be [illegible] on a factor of purity, i.e., the pulps with the highest attendant gas life [illegible] those pulps which probably have the least amount of water-soluble material. The other differences in the pulps most likely relate to freeness which in [illegible] cannot be adjusted by refining to a range that would be desirable for [illegible] board. Since the effect of refining was not investigated in this phase of the program, no relations can be concluded as to the effects of pulp treatment [illegible] the character of the pulp.

THE USE OF SIZING AND WET-STRENGTH AGENTS

Investigations into the effect of pH on gas life (Reports 5 and 6) showed that any acid conditions encountered after the addition of the ASC charcoal to a pulp slurry were detrimental to gas life. Consequently, the use of rosin or any other sizing material requiring acid conditions for the deposition of size on the pulp fibers was ruled out as impractical (the use of such materials could necessitate an extra step in the board-forming operation in which the pulp would have to be thoroughly washed after sizing in order to raise the pH). The field of possible sizing and wet-strength agents was, thus, narrowed to those materials which would be effective under alkaline conditions.

Aquapel

Aquapel is the designation of a series of alkylketene dimers manufactured from long-chain fatty acids by Hercules Powder Company. They are effective as sizing materials in the pH range of 7.5 to 8.5. In answer to our request for advice and samples of Aquapel, we received two samples of Aquapel 364 from Hercules (Aquapel 364 is derived from a mixture of 45% stearic acid and 55% palmitic acid). These samples consisted of Aquapel 364, a 6% emulsion of Aquapel 364 containing 3% starch, and Aquapel 486, which is a powder consisting of Aquapel 364 deposited on an equal weight of fine silica. The emulsion is intended for headbox additions or surface applications due to the tendency for the emulsion to "break" under mechanical stress. According to Hercules' recommendations, retention of either form can be improved through the use of a cationic starch (such as Cato 8) or Kymeme 557 (a wet-strength agent). A representative of the Paper Makers' Chemical Department of the Hercules Powder Company stated in a letter that very little work had been done with Aquapel and insulation board since it seemed difficult to size. He suggested that the board be overdried for 15 to 20 minutes at 250°F. or more and that some unbleached kraft be incorporated in the board.

Contact had been established with the Minnesota and Ontario Paper Company representatives and they offered to furnish us pulp for our experiments. However, the only practical way to furnish this in any quantity was to take the wet formed board from their commercial machine before entering the drier. This contained a small quantity of wax-rosin size. In order to compare the possible effect of this size on gas life or on other additives, a small quantity of similar stock before the addition of this size was dewatered by hand and also shipped to Appleton. A sample of the finished board made from this stock was also furnished.

The first work with Aquapel was carried out with the Minnesota and Ontario white board wet lap containing a small amount of wax-rosin sizing which had been washed in an effort to remove the rosin and alum (Reports 6 and 7). Enough residual size remained in the washed pulp so that board, formed from this pulp without additives other than charcoal, possessed a fair amount of water repellency. It was felt that the residual size in the pulp would introduce a confounding factor into the evaluation of any other sizing agents; consequently, the evaluation of the sizing materials was carried out with Wood Conversion Company pulps (Reports 7, 8, and 9).

The Aquapel 486 additions were made to 4% slurries of the pulp both before and after the charcoal additions. The Aquapel 360 additions were made in the deckle box in order to remove any possibility that the emulsion would be broken by the agitation of the Lightnin' mixer.

The effect of increasing amounts of Aquapel 364 (added as the active material in the form of either Aquapel 360 or Aquapel 486) is shown in terms of water absorption in Fig. 1 through 3. The additions are given as the per cent active material based on the ovendry fiber in the board. The boards treated with Aquapel 360 (Fig. 1 and 3) were formed from a pulp mixture of 52.2% Wood Conversion

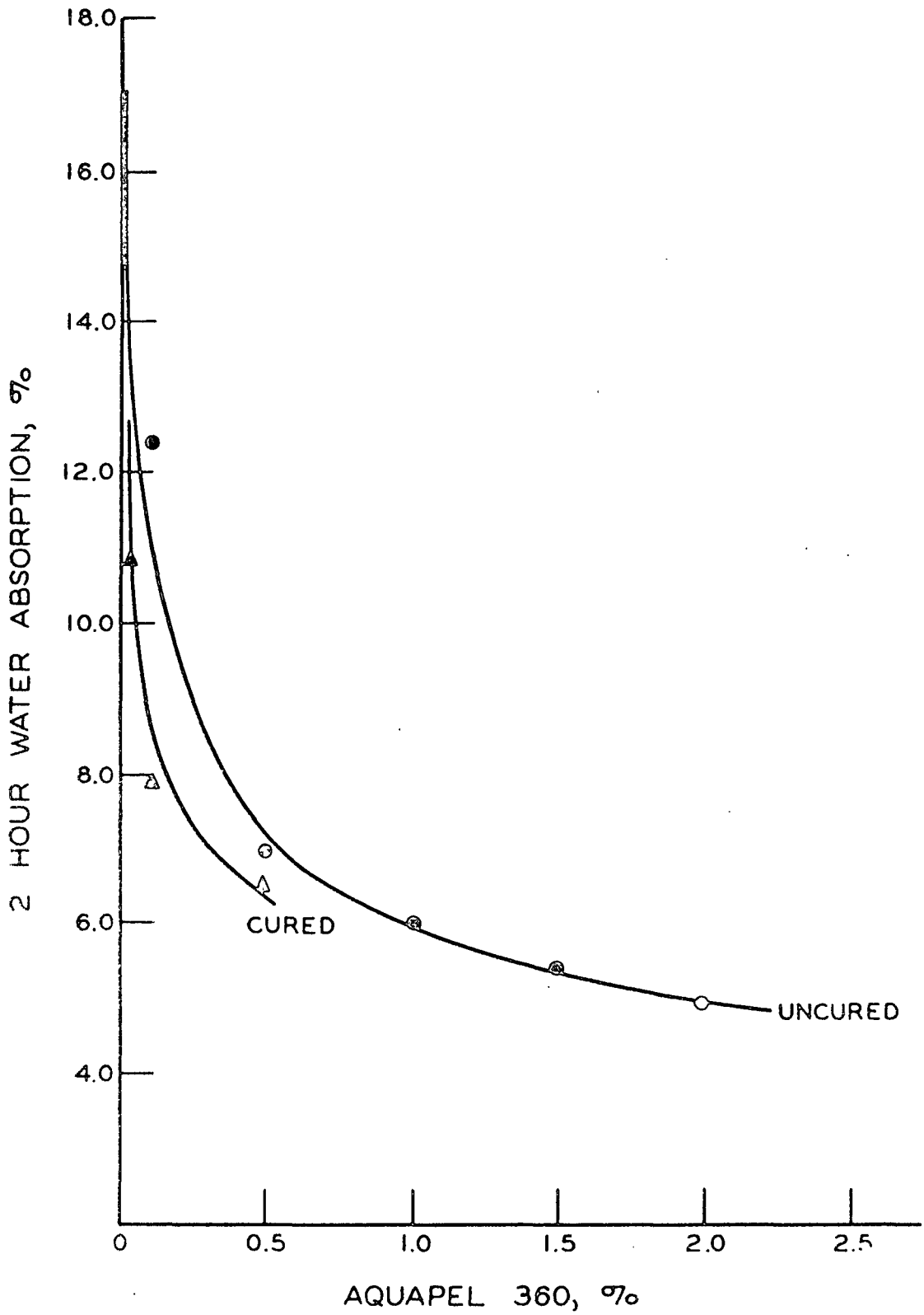


Figure 1. Water Absorption Curves for Board Treated with Aquapel 360

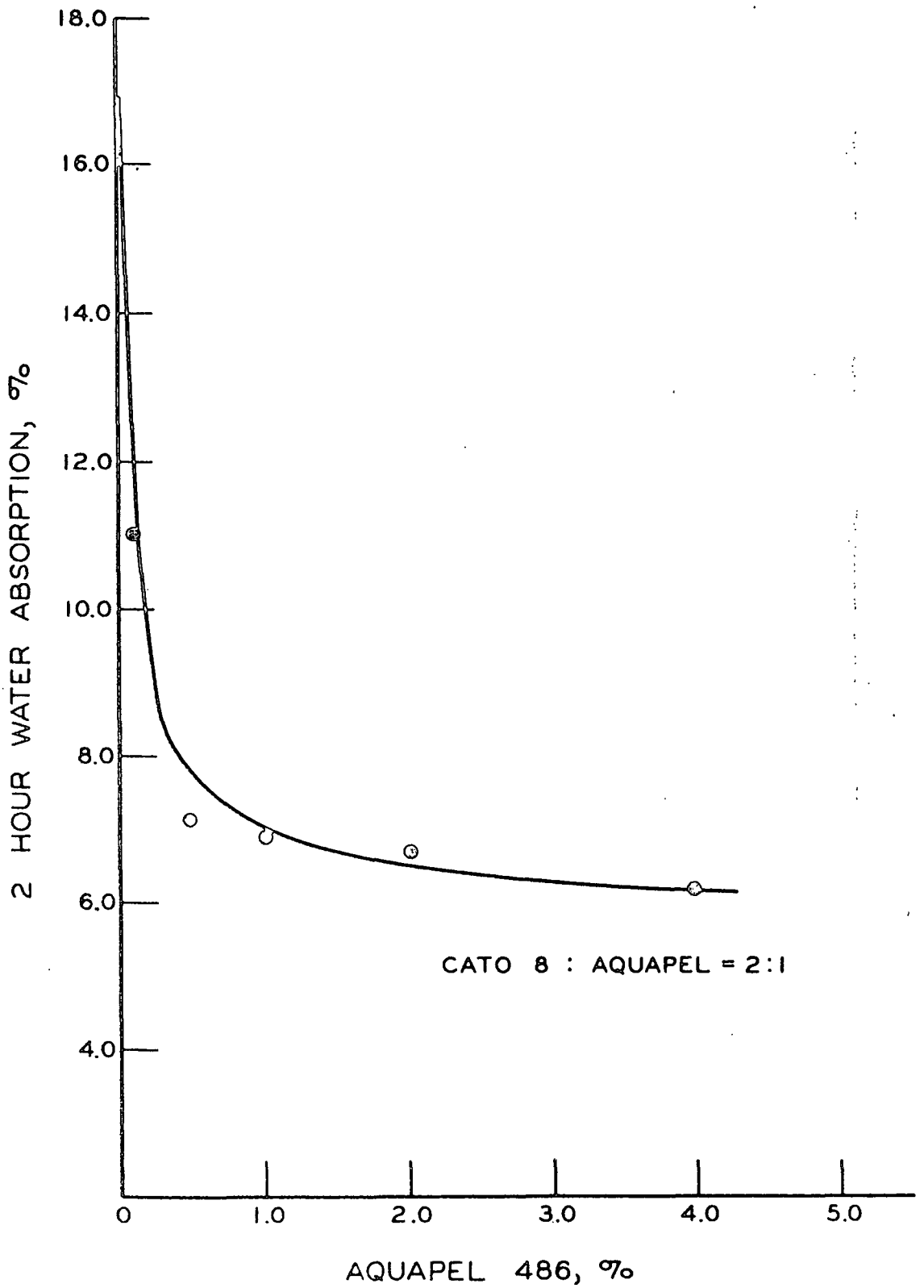


Figure 2. Water Absorption Curve for Board Treated with Aquapel 486 and Cato 8

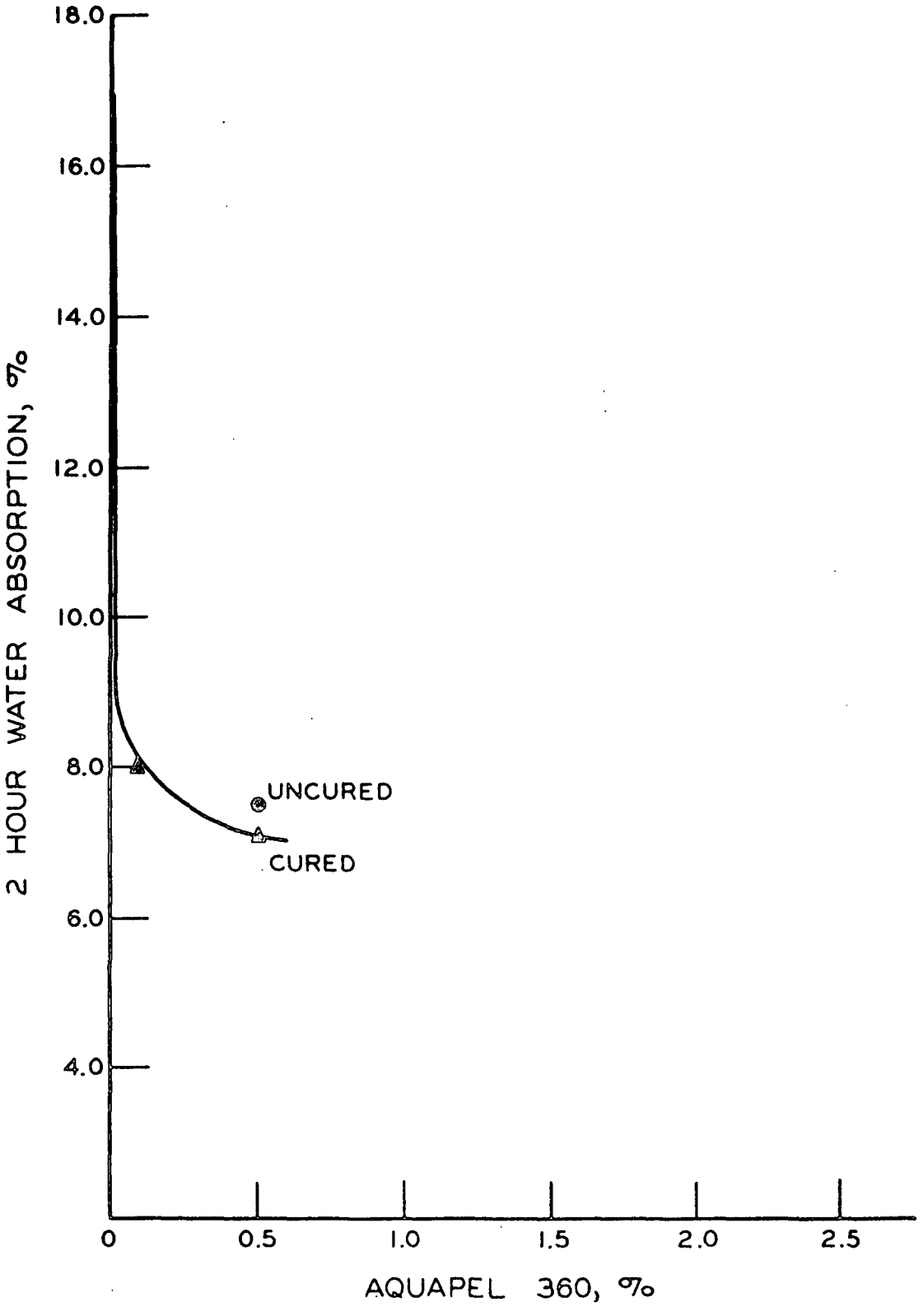


Figure 3. Water Absorption Curve for Board Treated with Aquapel 360 and 0.2% Kymene 557

Company 10-855 stock and 47.8% Wood Conversion Company 10-853 stock. Boards treated with Aquapel 486 (Fig. 2) were formed from a pulp mixture of 17% Wood Conversion Company 10-855 stock and 83% Wood Conversion Company 10-890 stock. All of these curves seem to approach a minimum water absorption of 5% by volume and level off at addition levels of 1.0% Aquapel.

Aquapel additions are plotted against cyanogen chloride gas life in Fig. 3 through 6. These curves show a rapid loss of gas life at additions of less than 1% Aquapel and indicate that Aquapel additions must be kept well below 1%.

Curing (overdrying at 300°F. for one hour) has a pronounced effect on water absorption at low addition levels; however, it also has a serious detrimental effect on gas life. Figure 1 shows that an uncured board containing an addition of .5% Aquapel 364 would absorb 7.2% water, the same absorption as a cured board containing 0.25% Aquapel addition; these boards would have the same gas lives according to Fig. 4.

Kymene 557

Kymene 557 is a wet-strength resin manufactured by the Hercules Powder Company for use in the pH range of 6 to 10. It is described as "a cationic water-soluble polymer with a Kjeldahl nitrogen content of 12.8% (dry basis)"--Hercules' description. This material is supplied in a water solution with a 10% solids content. It is recommended for use with Aquapel.

Cyron Size

Cyron size (American Cyanamid Company) is a thermoplastic water-dispersible, synthetic wax. It is obtained in a 100% active form to be used as a 5% dispersion in water. It is meant for use under alkaline conditions (pH range of 8 to 8.5 was used). This material was used for deckle box additions after the

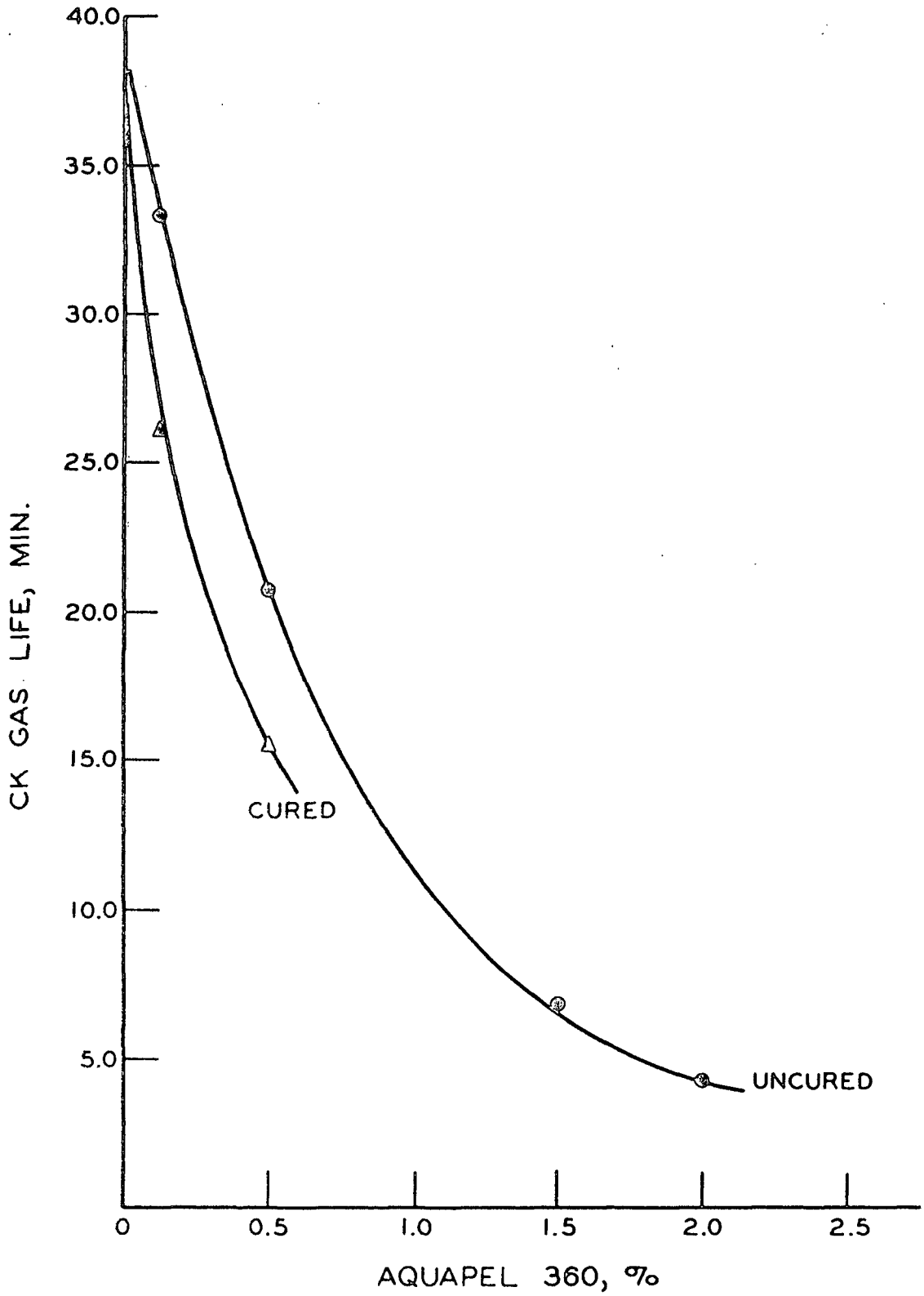


Figure ... Gas Life Curves for Board Treated with Aquapel 360

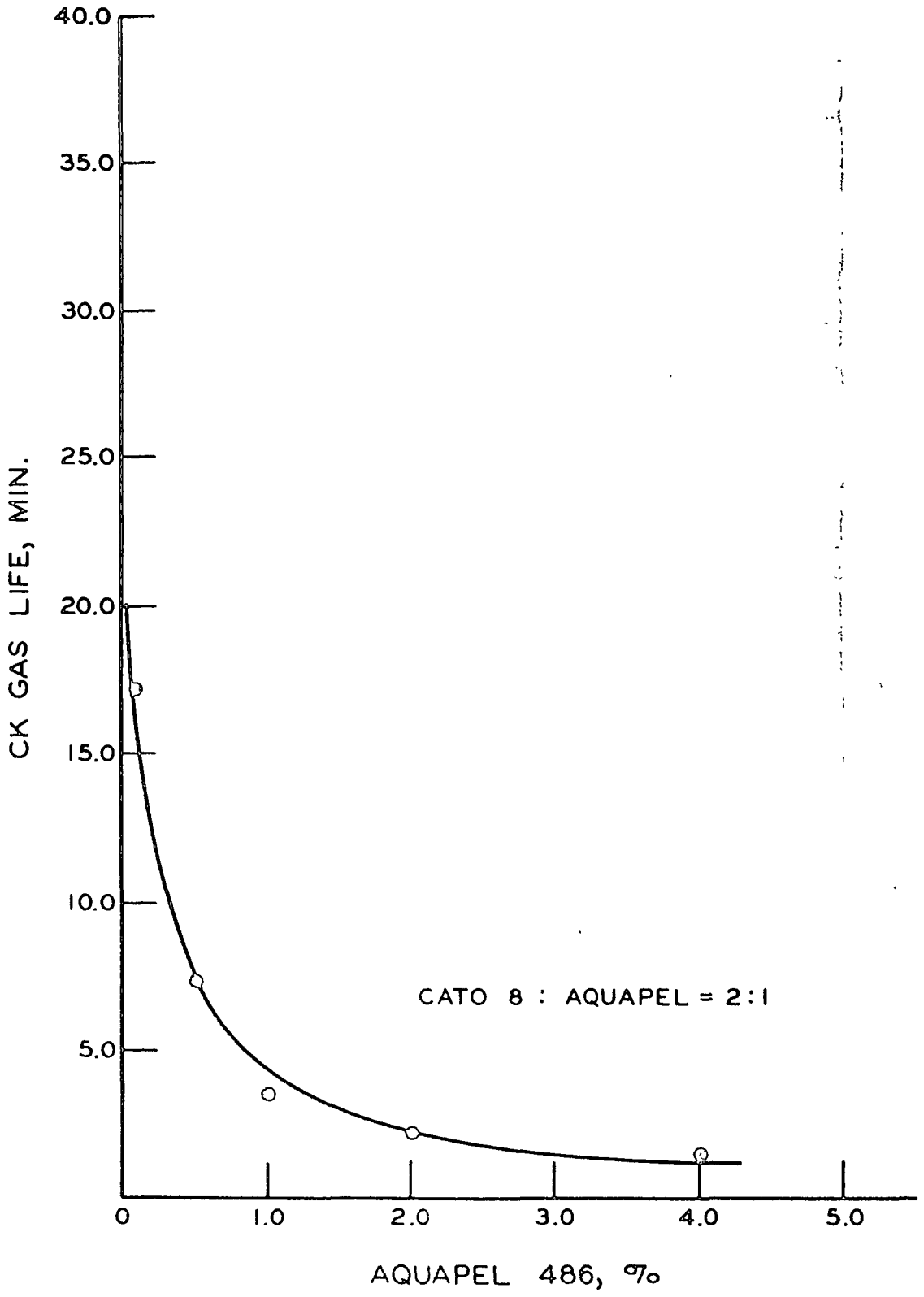


Figure ... Gas Life Curve for Board Treated ... Aquapel 486 and Cato 8

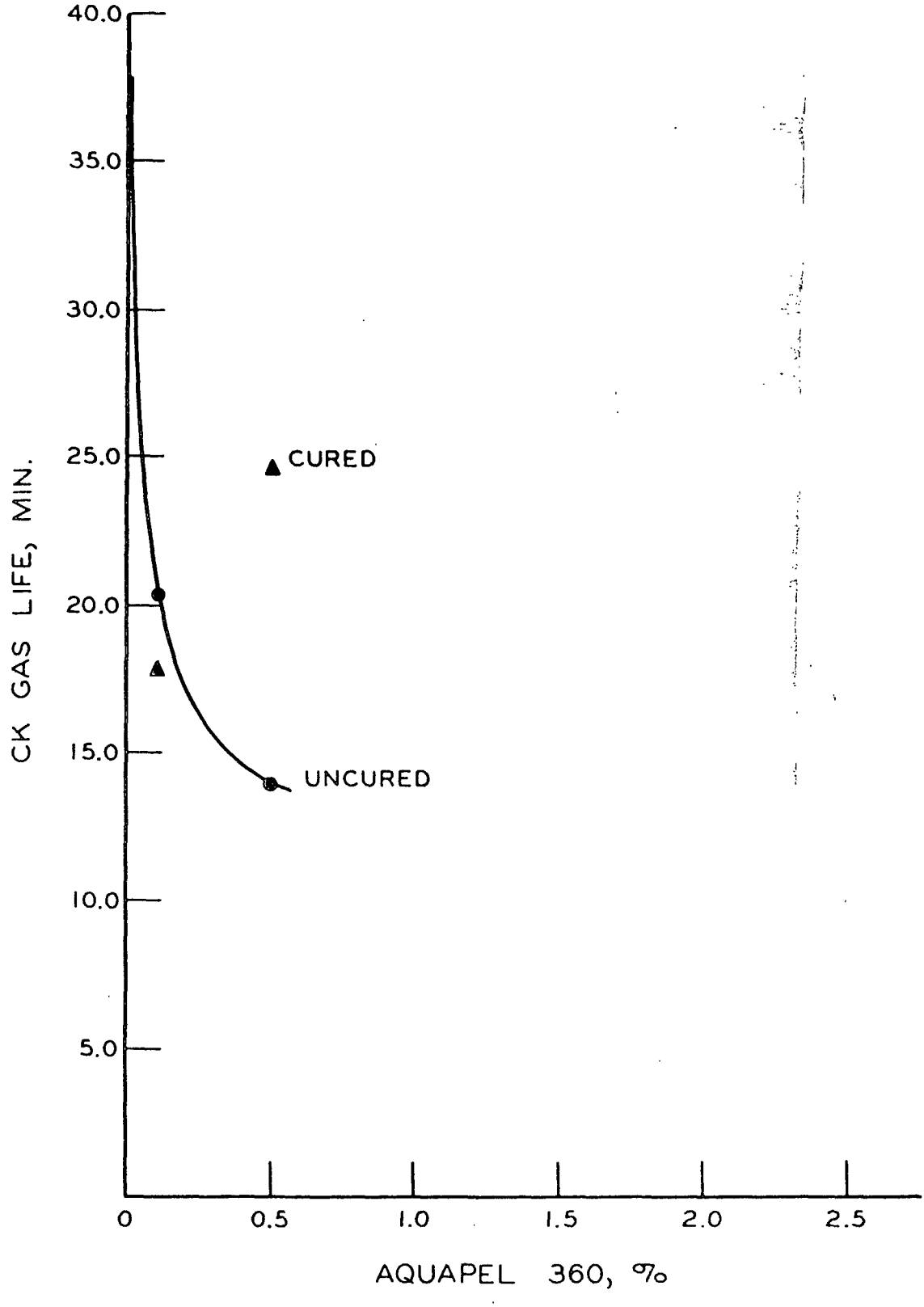


Figure 6. Gas Life Curve for Board Treated with Aquapel 360 and 0.2% Kymene 557

charcoal addition and for additions to the 4% slurry before the charcoal addition (Reports 7 and 8). A pulp mixture of 52.2% Wood Conversion Company 10-855 stock and 47.8% Wood Conversion Company 10-853 stock was used.

Use of Cyron in deckle-box additions increased drainage times somewhat and caused a large reduction in fiber bonding as evidenced by the increased caliper of the board relative to other board produced from the same pulp and the low strength of the board. Cyron additions before the charcoal produced the same results, but to a lesser degree.

The effects of Cyron additions before the charcoal additions on water absorption and gas life are shown in Fig. 7 and 8. According to these curves, a 2.25% Cyron addition is required to reduce the water absorption to 7%; however, an addition of this amount of Cyron reduces the cyanogen chloride gas life to 16 minutes. The use of this material would be a compromise of minimum properties in terms of gas life and water absorption.

Wet and Moisture Absorption

Conventional sizing systems protect primarily against liquid water and are not expected to change significantly the absorption of water vapor. An immersion test is an exposure to liquid water under a slight pressure and also an exposure to 100% relative humidity. In order to estimate the lowest immersion value that could be expected from a sizing treatment, tests were made of absorption by board when exposed only to high humidity.

Moisture isotherms were determined by measuring the moisture absorption, in grams, of samples conditioned to 75°F., 50% R.H.; 75°F., 92% R.H.; and 73°F., 97% R.H. Two specimens of each sample were equilibrated to each condition, three days allowed for equilibration. Before conditioning, each specimen was

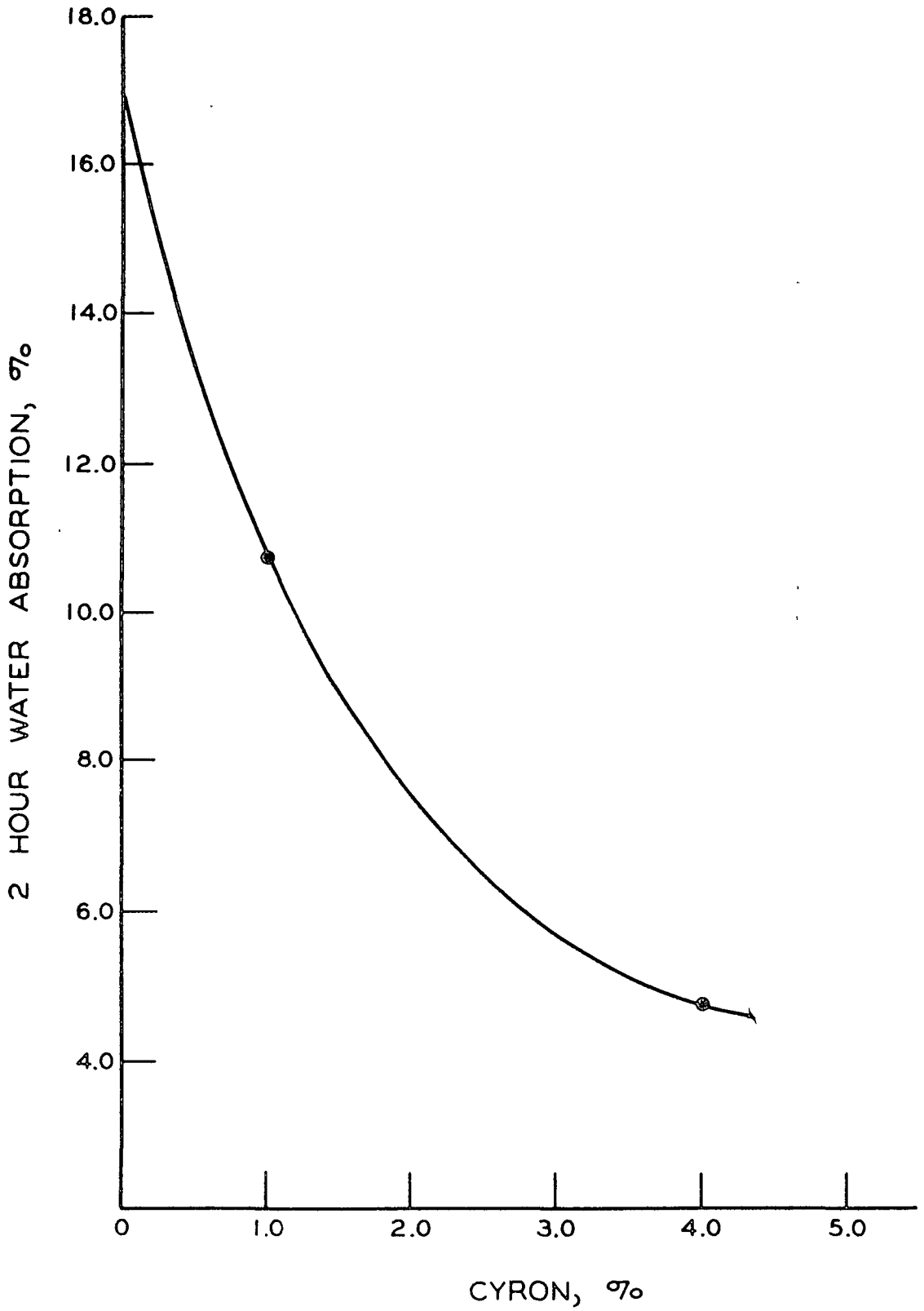


Figure 1. Water Absorption Curve for Board Treated with Cyron

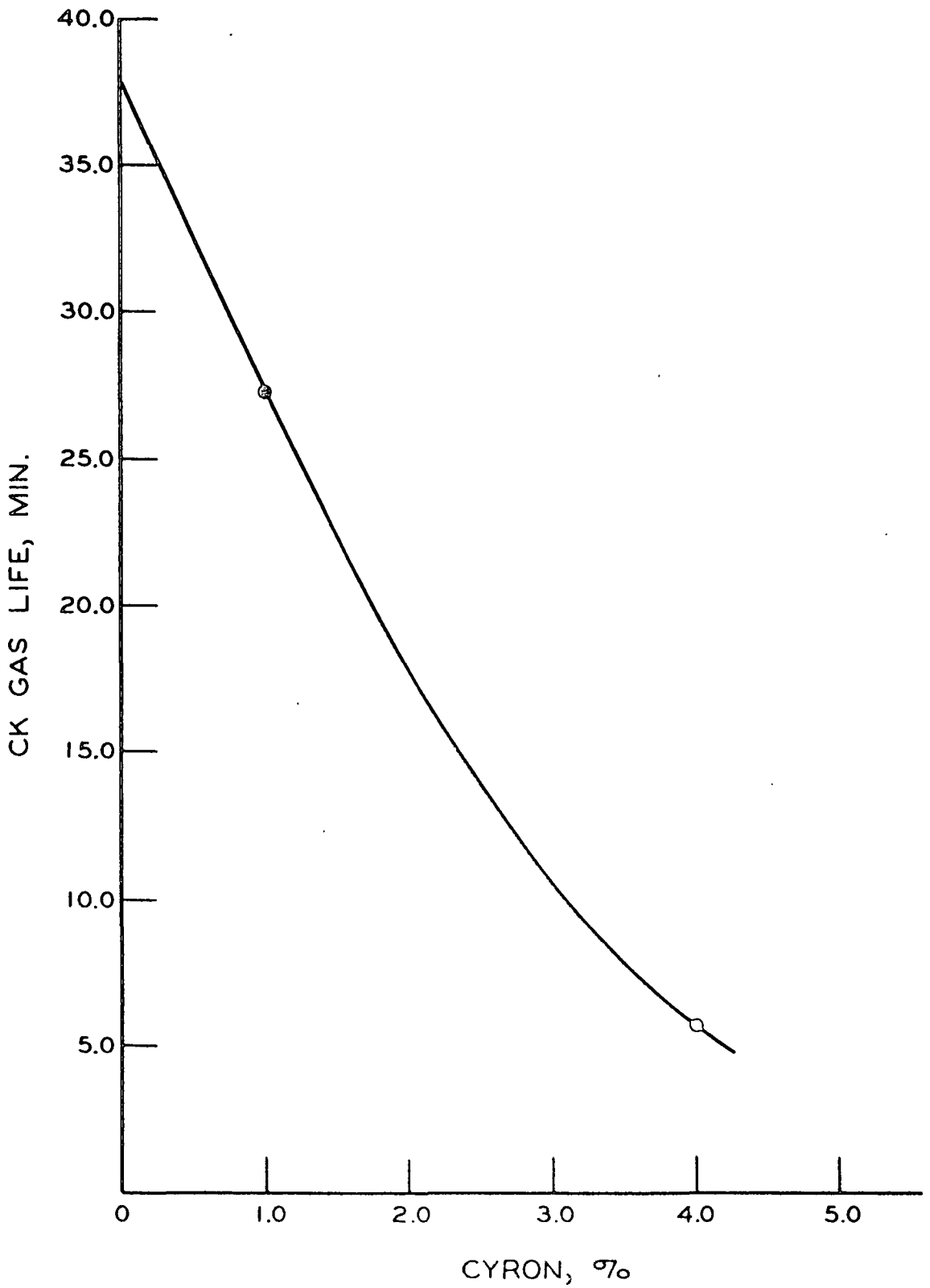


Figure 8. Gas Life Curve for Board Treated with Cyron

dried in an oven at 160°F. for 24 hours. The moisture content of each specimen was calculated by dividing the weight of the water absorbed by the dry weight of the sample.

Extrapolation of moisture isotherms for Aquapel-Kymene sized and unsized board samples indicated that both samples would absorb 28% water by weight (Report 11) or 9.5% by volume for a 21 lb./cu. ft. board. The Aquapel-Kymene size system produces resistance to penetration by liquid water but it does not physically seal the board (it is permeable to water vapor); consequently, the minimum absorption that could be expected for a sized board submerged in water at 73°F. would be 9.5% by volume after the board had become equilibrated. Twenty-four hour water absorption values as low as 13% were noted for Aquapel-Kymene sized board (Report 9, Table IV; Report 7, Appendix I).

THE USE OF FUNGICIDES

Mildew resistance tests, according to the direct inoculation procedure outlined in Federal Specification CCC-T-191b, Method 5751 "Mildew Resistance of Cloth; Direct Inoculation, Pure Culture, Non-Sterile Specimen Method," were found impractical due to the difficulty in producing mold growth on control (untreated) specimens of diffusion board. Consequently, it was necessary to resort to the modified procedure described in the section of this report entitled Testing Procedures. The following materials were evaluated as additions to unsized, charcoal loaded, boards.

Dowicide G, the copper precipitate of Dowicide G (copper pentachlorophenate), Tributyltin oxide, and Cunilate 2419 (a copper-8-quinolinolate emulsion), were evaluated by comparing the mildew resistance and gas lives of boards treated with these materials (Reports 6 and 8). All of the materials tested showed low mildew resistance or poor gas life in the treated board samples. However, the

TBTO (tributyltin oxide) and the copper-8-quinolinolate did show some promise. The gas life properties of board treated with TBTO were thought to have been affected by the acetone solvent media used to introduce the material into the pulp slurry; however, subsequent boards containing acetone additions equal to the volume of acetone added as a solvent for the TBTO showed no losses in gas life resulting from the acetone additions (Report 11).

Samples of three TBTO dispersions were obtained from the Metal and Thermit Corporation (Report 11). Boards were formed with 0.2 to 0.5% active material additions of these dispersions; however, in the interim between the production of these boards and their evaluation it was decided that the toxic characteristics of TBTO precluded its use in the diffusion board program (we were informed by a representative of the Scientific Chemicals Company that TBTO in concentrations greater than 0.03% is dangerous to handle).

Boards were formed with 1.0 and 2.0% additions of copper pentachlorophenate added as a sequestered solution. Boards were also sprayed with solutions of sequestered copper pentachlorophenate and copper-8-quinolinolate (Cunilate No. 2419). This work was done just prior to the expiration of the 1960-61 contract; consequently, no tests were made on these boards. This work is further discussed in Report 11.

EFFECTS OF PLANING

The effect of planing was investigated for two reasons: (1) to determine whether this board possessed a surface condition in the form of a skin which might hamper the diffusion properties of the board (the presence of such a skin was reported in previous work on development of a diffusion board at the Forest Products Laboratories) and (2) to evaluate planing as a means of controlling thickness in laboratory work and in commercial production. Planing was also used

in studies of the effects of board thickness and charcoal content on gas life and investigations of the possible stratification of the charcoal in the board. All of this work has been described in Reports 7, 8, 10, and 12.

Charcoal content determinations and smoke penetration tests were run on planed sections in comparison with unplaned sections of boards formed from Wood Conversion Company pulps (Report 8). In the smoke penetration tests the change in pressure drop at 1 liter/min. flow was consistent with the caliper change, indicating structural homogeneity and no gross skin effects in regard to diffusivity. The charcoal contents of the sections indicated that the wire side of the board might be slightly richer in charcoal than the top side, possibly due to migration of the charcoal during drainage. (As judged by color, the extreme surface layer of the board on the wire side is deficient in charcoal.)

Gas life tests on planed sections at various charcoal loadings were not conclusive (Reports 10 and 12). However, the results seemed to point to the possibility that some gas life activity would be lost as a result of planing; such an effect might be explained by assuming that the surface of the board is low in charcoal content and that this surface protects the richer laminae below from exposure to gases at the surface of the board which are more concentrated than the gases actually entering the board.

## THE USE OF STABILIZERS

A review of the mechanism of CK protection (see below) suggested that the following materials might enhance the stability of the board, either from the standpoint of their oxidation potentials or their possible value as pH buffers:

1. Zinc oxide
2. Calcium carbonate
3. Copper oxide
4. [illegible]

Diffusion boards were formed with and without sizing (0.5% Aquapel and 0.2% Kymene) incorporating various amounts of these materials in additions ranging from one to ten per cent of the dry fiber. The stability of these boards under tropical conditions was compared to the stability of untreated sized and unsized boards formed at the same time from the same pulp. This work was reported in Reports 12, 13, and 14.

Additions of triethanolamine and calcium carbonate did not improve CK stability; the triethanolamine increased the critical bed of the unaged board. The zinc oxide and the cupric oxide additions resulted in decreased critical beds both before and after aging; the critical bed of sized board containing a 1% addition of zinc oxide aged in tropical storage for one week was 4.0 g./100 sq. cm. compared to 5.1 g./100 sq. cm. for untreated sized board (Report 14, Table II) and a similar decrease was noted with the use of cupric oxide in unsized board. It was also noted that increasing the zinc oxide additions from 1.0 to 5.0% did not improve stability nor did an increase in cupric oxide additions from 2.0 to 10.0% improve stability; these increases seemed to reduce stability in comparison to the effects of the smaller additions. The conclusion drawn from this work is that the presence of small amounts of either cupric oxide or zinc oxide would enhance the CK stability of a diffusion board.

## THE VARIABLES AFFECTING GAS LIFE

### Effect of pH

Forming pH was found to have considerable effect on CK gas life and no discernible effect on PS life (see Reports 5 and 6). A series of boards formed from Minnesota and Ontario pulp at various pH levels showed a marked drop in CK life as the forming pH became acid. The same effect on CK life was noticed in

boards formed with repulped newspaper under acid and alkaline conditions but the PS lives of the same boards were unaffected.

An adjunct to this pH effect on CK life was carried out on boards which had been aged and in all cases but one showed very little CK activity (see Report 11). The pH's of the cold water extracts of these aged boards were measured and compared to the pH levels at which the boards were formed. Each of the boards had a slightly lower pH after aging; however, it was not known how this related to the actual pH of the unaged board since the forming pH's were measured when the pulp was in slurry form. The pH's of the aged boards were in each case greater than 7. While this does not indicate that pH is not a factor in ASC charcoal deterioration it does show that charcoal deterioration does not involve gross changes in pH that might affect the whole board.

The Critical Bed Concept

A typical plot of CK life versus charcoal loading for an undeteriorated board is shown in Fig. 9 (see also Report 10). This plot consists of a curved section, which develops into a straight line at a loading of approximately 3 g./100 sq. cm. The intercept of this straight line at 0 min. gas life is called the actual critical bed.

A series of plots of CK life versus charcoal loading for board samples containing charcoal in various stages of deterioration showed that, for samples having CK lives greater than 20 min., the effect of deterioration was to shift the straight-line portion without changing its slope (Report 10). Consequently, it appeared that boards varying in charcoal loading could be compared by extrapolating the CK life at a given charcoal loading along a straight line having a slope of 20.1 min./g./100 sq. cm. to zero minutes of CK life. This intercept would be an apparent critical loading; however, in this work it is referred to as the critical

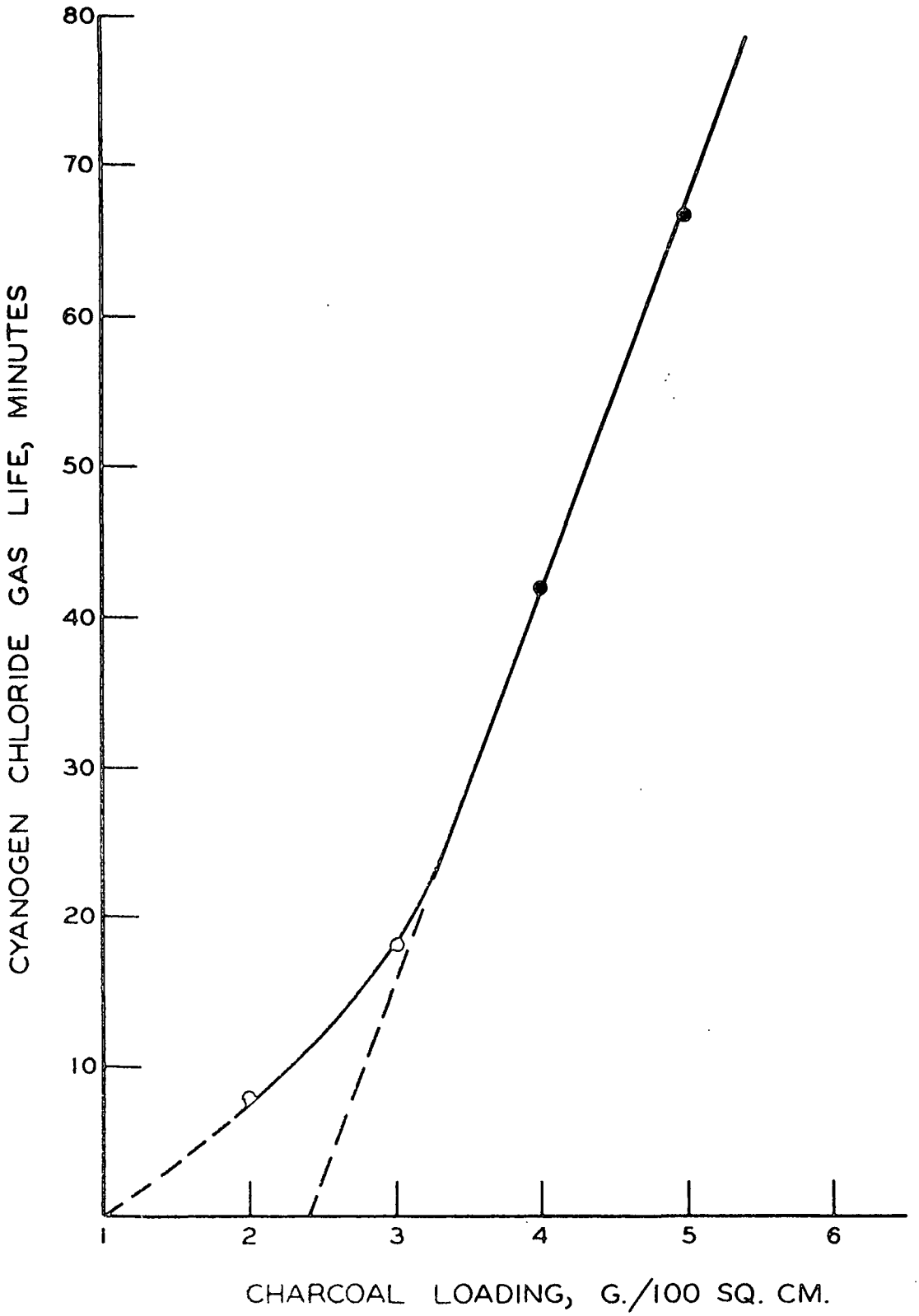


Figure 9. Plot of Charcoal Loading versus [illegible] for Aged, Unpoisoned Charcoal

A CK life--charcoal loading plot made up with data from a series of undeteriorated boards with charcoal loadings ranging from 1.7 to 6.7 grams was similar but not identical to the original (Report 12). However, the plot did show that the straight-line portion of the curve could be represented by a straight line with a slope of 26.5 min./g./100 sq. cm.

This concept was used as a means of comparing the CK lives of samples with varied charcoal loadings.

Aging

It was not until late in the program that aging in short-term storage was found to be a critical factor. A group of boards, tested for the first time after two months storage in ambient conditions, had CK gas lives somewhat below expectation. Retests of board samples were made and compared to the original CK lives of the samples. These retests confirmed the suspicions that aging for even short periods (at least under some conditions) could drastically affect CK life. Boards aged 65 to 180 days showed critical bed increases ranging from 0 to 22.5% (Report 10, Table I).

A planned program of accelerated aging was initiated in which boards were aged under tropical and desert conditions. Board specimens made for these tests were dried more thoroughly and sealed in polyethylene bags immediately after removal from the drying oven so that the effects of ambient atmospheric conditions would be mitigated. The specimens were unsealed immediately before being placed in an aging environment (Reports 11 and 12).

The gas lives of Aquapel-Kymene sized boards (0.5% active Aquapel 360 and 0.2% active Kymene 557) were compared with unsized boards formed from repulped newspaper, unwashed Wood Conversion Company pilot run pulp, and washed Wood

Conversion Company pulp before and after aging in tropical and desert conditions (Report 12, Table I). Aging under desert conditions produced no discernible effect on critical beds; tropical storage seriously decreased the CK protection in all boards, with the least effect in unsized boards formed from repulped newspaper. This indicates that moisture is a prime factor in deterioration; however, as evidenced by the relative stability of the unsized repulped newspaper boards, other factors also have a large influence.

There seemed to be an indication in the aging data and in the unaged CK life data for boards formed from washed and unwashed pulp that CK life deterioration might be greatly affected by certain pulp "impurities." A series of unsized boards were formed from a small batch of Wood Conversion Company pilot run pulp which had been "deresinated" by treating it with Igepal CO-630 (Report 13). Critical beds were determined for these boards before and after aging in tropical conditions and compared with the critical beds of aged and unaged samples of unsized boards formed from washed and unwashed Wood Conversion Company pilot run pulp (Report 14, Table III). The CK stability of boards formed from the deresinated and washed pulps was superior to that of the board produced from unwashed pulp, and the aged deresinated board had a lower critical bed than the unwashed pulp. In each comparison, however, the difference was slight, the critical bed after tropical aging being 4.6 g./100 sq. cm. for the unwashed pulp boards, 4.2 g./100 sq. cm. for the hot water washed boards, and 4.0 g./100 sq. cm. for the deresinated board, indicating that on a commercial scale the benefits of such treatments would be doubtful and expensive.

Chloropicrin (PS) gas life tests were run on the aged and unaged deresinated boards. The lives in both cases were 36 min. which is consistent with previous data indicating the PS life is not materially affected by aging. The

36-min. life was also equal to the PS lives of similar, untreated boards (see Report 12, Table II) indicating that the pulp treatment had no effect on PS activity.

Mechanism of CK Protection

Near the end of the year contract, recently declassified reports from the Office of Scientific Research and Development were received; these describe the impregnation procedure for the production of ASC charcoal, studies of the conditions and causes for deterioration, and the mechanism by which cyanogen chloride is removed. According to one of the reports, the ASC solution used to treat the charcoal contains 8.0% copper, 12.0% ammonia, 8.0% carbon dioxide, 2.0% chromium, and 0.2% silver; the copper, ammonia, carbon dioxide, and chromium are in the form of two complexes: $Cu(NH_4)_4CO_3$ and $Cu(NH_4)_4CrO_4$.

Both copper and hexavalent chromium are necessary for CK protection; as a result of some studies of the characteristics necessary for CK protection the following reactions were postulated in the report:

1. $5\ CHCl + 5H_2O \longrightarrow 5HOCN + 5HCl$
2. $2\ HCl + CuO \longrightarrow CuCl_2 + H_2O$
3. $3\ HCl + Cr^{+6} \longrightarrow 3/2\ Cl_2 + Cr^{+3} + 3H^+$

Here the $Cr^{+6}$ acts as a hydrolysis catalyst and then reacts with the hydrochloric acid in Step 3. In the deterioration studies described in the report it was found that the deterioration of ASC charcoal stored in atmospheres of carbon dioxide and ammonia was greater in the carbon dioxide atmosphere. This was attributed to the effect of carbon dioxide on pH, the increased hydrogen ion concentration causing the hexavalent chrome to be reduced to the trivalent state.

## PILOT TRIALS

A series of pilot trials were conducted at the Bauer Brothers Company in Springfield, Ohio on May 15 through May 17, 1961. These trials were described in Reports 8 and 9; a copy of the Bauer Brothers report was included in Report 9.

The trials were made with stock obtained from the Wood Conversion Company, consisting of a blend of two separately refined stocks. Part of the stock was further refined at Bauer Brothers and blended with the "as received" stock.

Runs were made with the following additions to the fiber:

| Run | Additions |
|---|---|
| 1 | 25% charcoal |
| 2 | 25% charcoal followed by 0.5% (active material) Aquapel 360 |
| 3 | 25% charcoal followed by 0.5% (active material) Aquapel 360 followed by 0.2% (active material) Kymene 557 |
| 4 | 0.5% (active material) Aquapel 486 followed by 25% charcoal, followed by 1.0% Cato 8 |
| 5 | 25% charcoal followed by 1.0% Cyron |

Samples of the boards produced in these trials were tested at the Institute for carbon dioxide diffusivity, water absorption, ash, smoke penetration, and physical strength and at the Army Chemical Center for gas life.

The ash contents of the boards were high and inspection of pulp samples sent to the Institute showed the presence of some glassy material in the pulp, possibly mineral fiber contamination from previous runs at Wood Conversion Company.

The range of values of some of the tested properties of the pilot run boards are shown in Table III. Complete results of these tests were presented in Table IV of Report 9. The protective properties were adequate as defined at the Army Chemical Center (see Introduction), the density range was slightly lower than the contract specification, and sizing markedly decreased water absorption but did not seem to seriously hamper the other properties.

TABLE III

GENERAL SUMMARY OF PROPERTIES OF PILOT RUN BOARD

| Test | Range |
|---|---|
| Density | 16.8 to 19.9 lb./cu. ft. |
| $CO_2$ diffusivity | 2.95 to 3.66 x $10^{-2}$ sq. cm./sec. |
| Water absorption, 2 hr. | 5.04 to 6.40% (by volume) for sized boards<br>20.24 to 28.88% (by volume) for unsized boards |
| Water absorption, 24 hr. | 12.18 to 14.05% (by volume) for sized boards<br>36.87 to 46.14% (by volume) for unsized boards |
| Smoke penetration | Satisfactory resistance in all boards |
| Gas life, cyanogen chloride | 23.2 to 43.4 min. |
| Gas life, chloropicrin | 24.3 to 32.5 min. |

The minimum specifications for Class E sheathing board (outlined in Federal Specification LLL-I-535 "Federal Specification for Insulating Fiberboard") may be used as one guide to the properties required for a board similar to diffusion board in structure and use. This specification requires dry tensile strength of 150 p.s.i. and a minimum transverse loading strength of 12.5 lb. total load on a 10-in. span, 3-in. wide and 1/2-in. thick (equal to TAPPI Standard Modulus of Rupture of 300 p.s.i.). The range of values obtained in the strength tests on the pilot run boards are shown in Table IV (complete results were presented in Table V of Report 9). For the most part these values surpassed the minimum specifications of the Federal Specification. The effect of sizing, was a substantial

improvement in wet strength and some improvement in dry strength. Of the sizing materials the Cyron seemed to produce the least effects while the Aquapel-Kymene system produced the greatest effects, particularly on wet strength.

TABLE IV

GENERAL SUMMARY OF STRENGTH PROPERTIES OF PILOT RUN BOARD

| Test | Range |
|---|---|
| Modulus of rupture | 256 to 481 p.s.i. |
| Dry tensile | 233 to 452 p.s.i. |
| Wet tensile, 2 hr. soak | 13 p.s.i. for unsized board<br>111 to 214 p.s.i. for sized board |
| Wet tensile, 24 hr. soak | 10.5 p.s.i. for unsized board<br>60 to 115 p.s.i. for sized board |

In general, the board sized with Aquapel 360 and Kymene 557 (Run 3) had the best gas life and physical characteristics. The gas lives of boards produced in Run 2 were lower than the gas lives of boards produced in Run 3 and in most cases the strength of boards produced in Runs 2 and 4 were lower than Run 3. All other tested characteristics were comparable for these runs.

# PRODUCTION PLANNING

## DRYING STUDIES

Drying studies were carried out in order to determine the rate of moisture loss in the board and the temperatures encountered in the board during the drying process at various oven temperatures. The specimens dried in these trials were evaluated in terms of CK gas life so that any special conditions or restrictions necessary to the preservation of CK activity in a commercial operation could be anticipated. These studies were described in Report Nine.

Oven temperatures as high as 425°F. were used and temperatures as high as 320°F. were encountered on the surfaces of the specimens without detriment to the CK activity of the board. The internal temperature of each specimen tested reached 220°F. as this was used as the criterion of total dryness. The conclusion drawn from these studies was that this board could be dried in a zone drier at temperatures limited by the scorch temperature of the board.

## FLAMMABILITY

Flammability tests were conducted with specimens from the Bauer Bros. pilot run and boards formed at the Institute from the pilot run pulp without charcoal additions (Report 9). These tests were made to determine the effect of charcoal on the thermal stability of this board and the possible fire hazard in commercial driers.

### Ignition Temperature

A thermocouple was inserted into a specimen, thermocouples were attached to both surfaces of the specimen and the specimen was placed in a circulating air oven. The temperature of the oven was increased in increments of ten degrees,

increasing the oven temperature after the specimen had become equilibrated to the oven temperature. The temperature at which the specimen temperature first exceeded the oven temperature was called the ignition temperature. Temperatures were recorded on a recording potentiometer.

Specimens of charcoal-loaded boards were badly charred after five minutes exposure at 410°F. in a circulating air oven, while blank specimens were only mildly scorched after 2 hrs. exposure at 410°F. The charcoal-loaded boards ignited within one minute when exposed to 430°F. and the blank boards required three minutes exposure at 500°F. for ignition.

Flame Test

This test was carried out in accordance with the specifications for the Inclined Panel Flame Test as outlined in the ASTM specification C-209-60 "Testing Structural Insulating Board Made From Vegetable Fibers." In this test a specimen, 12 by 12 inches is supported at an angle of 45° and ignited by burning 1 ml. of anhydrous ethyl alcohol at a point one inch below the bottom surface of the supported specimen and 3 inches in, centered, from the lower edge measured along the bottom surface of the specimen.

The test was carried out under ambient conditions in a draft-free hood. Burning or glowing boards were extinguished one minute after exhaustion of the fuel by placing them in a container of gaseous carbon dioxide. Results of the tests were reported as the area of char computed from the area of an ellipse having major and minor axes equal to the maximum length and width of the charred area. The specimens were supported on four one-half inch dowels tapered to 1/8-inch points in order to provide maximum access of air for combustion.

Tests on charcoal-loaded specimens resulted in charred areas of approximately 30 sq. in. These specimens tended to glow rather than flame. The blank

specimen (no charcoal) tended to flame and no glow was observed; the charred area for this specimen was 40 sq. in. Apparently, the addition of charcoal reduces the ignition temperature and produces a tendency for glow rather than flame.

NEGOTIATION FOR PRODUCTION RUN

Inquiries were addressed to a number of companies producing insulation board to determine which companies might be interested in co-operating in a production run of 15,000 sq. ft. of board. Several companies indicated that they would be unable to co-operate because of fiber and/or equipment limitations.

The Minnesota and Ontario Paper Company indicated that they would co-operate in a production run, using their regular insulation board grade of groundwood without additional washing.

The Wood Conversion Company offered to co-operate in such a run utilizing their lightly cooked aspen fiber (similar to the fiber furnished for the Bauer Bros. pilot run), suitably refined, with additional washing if desirable. They expressed a preference to run at a thickness of 3/8 inch and also suggested a preliminary trial of 1000 sq. ft. on an available machine which is full scale but lacking in drier capacity for full-scale production. The preliminary trial would minimize the necessity for adjustments during the production run on the more expensive commercial unit.

The Simpson Timber Company indicated that they would be willing to co-operate. They described their equipment which includes a pilot machine without a drier. The fibers most easily available in their operation are softwoods consisting of Douglas-fir and western hemlock. Samples of their fibers were requested as they had not been investigated.

## GENERAL DISCUSSION AND PRESENT STATUS

From the results reported here it appears that there should be no difficulty in producing on a commercial scale a diffusion board which will meet the tentative requirements for diffusivity, resistance to aerosol penetration, and resistance to penetration of toxic gases as represented by chloropicrin. Considerable improvements in wet strength and water repellency have been obtained and the strength of the boards produced on a pilot scale exceeds the requirements covered in Federal Specifications for structural fiber insulating board. Evaluation of resistance to mildew is difficult to evaluate. By the method originally proposed even the boards without addition of special fungicides showed very little growth. Based on commercial experience it is probable that additional resistance to mildew can be obtained by use of conventional materials but these may affect gas life.

The critical feature of the board production appears to be the retention of resistance to the passage of cyanogen chloride. Variations in pulp quality and the addition of wet strength, sizing agents, or fungicides all seem to have a detrimental effect on this property. In the work reported here it appeared that a reasonable balance between wet strength, water repellency, and mildew resistance, on the one hand, and CK gas life on the other, could be obtained for a diffusion board. The resistance to CK penetration was not affected by accelerated aging at desert conditions but was seriously affected by tropical aging. It is known that the charcoal itself is somewhat sensitive to moisture, carbon dioxide, and similar materials and it appears probable that ASC charcoal will always be sensitive to the tropical deterioration even without the factors of pulp quality or additives.

The anticipated production run was delayed because of this question of tropical deterioration of CK life. Two proposals were submitted with the choice based on the importance of CK protection. One was for a production run to

duplicate on a commercial scale the relative balance of properties obtained in the laboratory and pilot run. The alternate proposal was for a research program aimed at investigating the mechanism of CK protection and clarifying the factors which control it. A contract has now been signed for an extension of the original process study and for two commercial runs for the purpose of obtaining as good a balance of properties with a minimum sacrifice of the presently available protective features but without emphasizing a research investigation of CK protection itself.

THE INSTITUTE OF PAPER CHEMISTRY

L. E. Leporte

L. E. Leporte, Research Aide

T. A. Howells

T. A. Howells, Chief
Special Processes Section